Phenolic buffer: R-OH + NaOH + H_2O pKa ≅ 9.4-I 0.8

Carbonate buffer: $Na_2CO_2 + H_2O \rightarrow NaHCO_3 + NaOH$ pKa ≅ 10.2

The high alkalinity is largely responsible for solubilizing the various organic constituents. If the pH is reduced, various organic constituents will precipitate, beginning with the components with low pKa values (e.g. the phenolics) and eventually those with higher pKa values (e.g. the carboxylic acids). Thus, the soluble component would vary as pH is reduced. Consequently, if the pH is adjusted in order to perform certain tests, the nature and composition of the test material will necessarily change.

C. Lack of Analytical Method

There is no analytical methodology available to measure spent pulping liquor. Spent pulping liquor is a mixture of numerous known, tentatively identified and unidentified components, and thus only some components would be available for calibration purposes. Given the complexity of the mixture, it is not currently possible to characterize spent pulping liquor as necessary to undertake a number of the SIDS endpoints. The HPV program does not encompass the kind of research program that would be necessary in order to develop an appropriate analytical method with sufficient sensitivity, if indeed it is even possible to do so. Consequently, because of the lack of an appropriate analytical methodology and the practical impossibility of developing such a method, many of the required SIDS endpoints that are part of the HPV Challenge program cannot be undertaken.

III. Review of Existing Data and Development of Test Plan Rationale for SIDS Endpoints

A. Physicochemical Data

Except for pH, physicochemical data for spent pulping liquor that satisfy the data evaluation criteria described in EPA guidance documents were not found. Most of the required physicochemical properties tests in the SIDS battery are designed for a single, pure chemical. Due to the fact that spent pulping liquor is an extremely complex mixture of inorganic and organic constituents, many of the common physicochemical parameters are inapplicable.

If one were to attempt these tests, the numerous different constituents in spent pulping liquor will respond to most physicochemical tests differently. The resulting wide ranges of values for the individual constituents would not represent the characteristics of the mixture.

The problem is further exacerbated by the lack of a suitable analytical procedure to measure spent pulping liquor. Absent a suitable anaiytical method for

measuring the spent pulping liquor, some of the tests cannot be performed. For these reasons, many of the SIDS physicochemical tests in this category cannot be performed or would not produce useful pKa information. Tests that are inappropriate for this material include pKa, water solubility, octanol-water partitioning coefficient (K_{ow}), and adsorption/desorption to soil. AF&PA will test the boiling point and vapor pressure of spent pulping liquor. Data on pH of spent pulping liquor are already available, and determination of the melting point is not necessary since the material is a liquid. The following narrative explains the rationale for this testing plan in more detail.

1. Water Solubility

Spent pulping liquor is a complex mixture of inorganic and organic salts suspended or dissolved in water. A test for water solubility could be performed on the test material, but it would result in multiple values for individual constituents. Due to the lack of a suitable analytical method for the complex mixture, it is not feasible to measure the water solubility for the mixture.

As noted above, strong black liquor contains 50-70% solids. At solids contents below 50%, the inorganic salts contained in spent pulping liquor are completely dissolved in the aqueous portion of the liquor. Often, the 50% solids point (the point where the salts start precipitating) is referred to as the "solubility limit." At solids levels greater than 75%, Burkeite ($2Na_2So_4 \cdot Na_2CO_3$) is the only salt that precipitates. Thus, between 50 to 75% solids, spent pulping liquor is essentially a water/organic-inorganic suspension (Adams et al. 1997).

2. Melting Point and Boiling Point

Because spent pulping liquor is a liquid under normal conditions, it is not necessary to determine the melting point. However, AF&PA will test to determine the boiling point.

3. Octanol:Water Partition Coefficient

Given the numerous organic and inorganic constituents in kraft black liquor, any assay used to estimate the partitioning properties would yield a range of values reflecting this complex mixture. Such values would be meaningless and would provide little, if any, useful information concerning the material. Consequently, the K_{ow} will not be conducted on this mixture.

4. pH

Already available data show that the pH of kraft black liquor ranges from 11.5 to 13.5 (various company Material Safety Data Sheets).

5. pKa

Because pKa determinations apply to specific compounds, this endpoint cannot be conducted on spent pulping liquor, which is a complex mixture.

6. Adsorption/Desorption to Soil

Due to the fact that black liquor is an extremely complex mixture of inorganic and organic constituents, the test for adsorption/desorption to soil would have little, if any, meaning. The different constituents will adsorb/desorb to soil differently, resulting in a wide range of values. Moreover, because there is no analytical method for the spent pulping liquor mixture, the adsorption/desorption to soil of spent pulping liquor will not be determined.

7. Density

The density of spent pulping liquor will be determined.

Summary: The boiling point, vapor pressure, and density of kraft black liquor will be determined. Data are already available on pH. Testing will not be conducted for pKa, water solubility, octanol-water partitioning coefficient, or adsorption/desorption to soil.

B. Environmental Fate & Pathways

Data on environmental fate for spent pulping liquor that satisfy the data evaluation criteria described in EPA guidance documents were not found. Described below is the feasibility of conducting the required SIDS testing for the fate and transport endpoints.

1. Photodegradation

The practicability of performing this test is hindered by the lack of an analytical procedure to measure spent pulping liquor. A test of photodegradation cannot be performed, since the composition and quantity of the test material before and after exposure to sunlight cannot be measured.

2. Hydrolysis

With respect to the hydrolysis test, the required test (OECD 111) is designed to measure hydrolysis (stability in water) of pure compounds at several pH levels (4-9) that are likely to be found in the environment. Thus, the test is not applicable to the alkaline, complex mixture of spent pulping liquor. In addition, this endpoint cannot be measured since an analytical method for spent pulping liquor is not available.

3. Biodegradation

An additional problem is presented for biodegradation testing. The high pH of the test material would not be compatible with survival of the bacteria, thus preventing the possible degradation of the material. However, the test guidelines allow neutralization of materials in order to conduct this test. While neutralization will alter the composition of the test material because various constituents will precipitate out as the pH changes, the test can be performed. An analytical method for spent pulping liquor is not necessary. AF&PA therefore proposes to conduct biodegradation testing, even though the results must be interpreted with caution.

Summary: Due to the complex nature of spent pulping liquor and the attendant lack of a practical analytical methodology for spent pulping liquor, the hydrolysis and photodegradation tests cannot be performed. Biodegradation testing will be performed after the test material is neutralized, although results will likely be of limited relevance.

C. Ecotoxicity Tests

Data on the SIDS ecotoxicity endpoints (acute toxicity to fish and aquatic invertebrates and toxicity to plants) that satisfy the data evaluation criteria described in EPA guidance documents were not found for spent pulping liquor.

The Animal Welfare Act does not apply to the aquatic test organisms. However, each of the ecotoxicity endpoints must be tested within a narrow pH range (6.5 to 8.5), consistent with maintaining the viability of the test organisms. Due to the high pH of black liquor (i.e., approximately 11.5 to 13.5), the only way that ecotoxicity tests could be conducted would be to neutralize test solutions to the lower pH range.

The latest OECD (#203) guideline suggests that adjustment of the pH with simple acid or alkali (or other suitable buffer) can be done, even though *"this can cause sedimentation and/or degradation of the test substance."* Adjustment of the pH can be carried out in the stock solution or in the media itself, as judged appropriate.

However, in the particular case of spent pulping liquor there is a further complication. Reducing the pH to levels at which the test organisms survive will effectively alter what is in solution. Consequently, whatever constituents remain in the aqueous phase, the resulting mixture would no longer be representative of spent pulping liquor.

Nevertheless, even though the "neutralized" material would not be representative of the chemical CAS number being tested, it could potentially represent a situation in which a spill of the very caustic material is diluted to a lower pH by virtue of accidental discharge into a large water body. Such a scenario is

unlikely. The resulting data would be of limited relevance. However, the test can be accomplished, and OECD guidelines contemplate using neutralized test materials.

Thus, even though the relevance of the results will be highly limited with respect to the potential ecotoxicity of spent pulping liquor, the SIDS ecotoxicity endpoints will be determined to fulfill the spirit of the HPV program.

Summary: Following appropriate adjustments of the pH of spent pulping liquor, this material will be tested for toxicity to fish, daphnia, and algae consistent with the required SIDS ecotoxicity endpoints. Data should be interpreted with caution, however.

D. Human Health Effects

Data on the SIDS human health effects endpoints of acute toxicity, genetic toxicity, repeat dose toxicity, reproductive and developmental toxicity for spent pulping liquor that satisfy the data evaluation criteria described in EPA guidance documents were not found.

However, with the exception of the *in vitro* tests for mutagenicity in Salmonella bacteria and mamallian cells, all of the other human health effects endpoints require the test substance to be administered to animals either by gavage or in the diet. The high pH of the test material in this case would result in immediate corrosive effects in the animals. Not only would useful mammalian toxicity data not be obtained, but the spirit of the HPV program requires that testing in which animals would suffer should not be conducted. Therefore, AF&PA will limit health effects testing to the mutagenicity endpoints and not perform mammalian toxicity tests for spent pulping liquor.

1. Likely Corrosive Effects

Given the extremely high pH of kraft black liquor (approximately 11.5-13.5), it would be impossible to administer such a caustic material to test animals without causing them to suffer. It is well established that highly alkaline material can cause chemical burns. *'Extremely corrosive* and *reactive chemicals may produce immediate coagulative necrosis that results in substantial tissue damage. . . ."* (Casarett & Doull 1997) As a leading occupational medicine text notes: *"Alkalis not only coagulate tissue protein by dessication or salt formation, but they also saponify fats and cause liquefaction necrosis."* (Zenz 1994) The severity of the effect will depend on the corrosiveness of the chemical. (Olishifski 1974). OECD's Guidance Document on the Recognition, Assessment, and Use of Clinical Signs as Humane Endpoints for Experimental Animals Used in Safety Evaluation provides that *"If something is known to* cause *suffering in humans, it should be assumed to* cause *suffering in animals."* (OECD 2000).

With a pH in the range of 11.5 to 13.5 (and with the strong black liquor test material generally expected to be at the higher end of this range), spent pulping liquor is clearly corrosive. When shipped, spent pulping liquor is labeled as corrosive (UN1 760 label) under Department of Transportation regulations. Manufacturers of spent pulping liquor comply with OSHA's Hazard Communication Standard, including providing Material Safety Data Sheets for the material. (OSHA defines as corrosive and therefore hazardous those chemicals that cause visible destruction of tissue at the site of contact. (Code of Federal Regulations, OSHA). EPA automatically defines waste as hazardous due to the characteristic of corrosivity if the pH of the material is 12.5 or higher. (Code of Federal Regulations, EPA).

Thus, based on well-known characteristics of any corrosive material, one would expect spent pulping liquor to result in chemical burns. Whether by gavage or via administration in the diet, the high pH of spent pulping liquor is expected to cause severe ulcerations or necrosis at the point of contact, i.e., esophagus or gastric mucosa, when fed to test animals.

2. Pertinent OECD Testing Guidelines

OECD guidelines provide that testing not be carried out when it will cause distress to the animals based on corrosive effects of the test substance:

- As noted in the guidelines for acute toxicity testing (OECD 401), "Dosing test substances in a way known to cause marked pain and distress due to corrosive or irritating properties need not be carried out." Indeed, OECD is currently taking steps to eliminate acute (LD_{50}) testing in light of animal use concerns.
- OECD 420 on acute toxicity further notes that, "doses that are known to cause marked pain and distress, due to corrosive or severely irritant actions, need not be administered."
- Moreover, OECD 422 governing repeat-dose testing provides: *dose /eve/ should be chosen with the aim of inducing toxic effects but not death* nor *obvious suffering."* [Emphasis added] It is not clear that these dual requirements can be satisfied simultaneously with such a corrosive material.

For spent pulping liquor, even small doses would likely result in *"obvious suffering"* of the test animals.

3. Animal Welfare Act and Other Licensing Provisions

The Animal Welfare Act, 7 U.S.C. § 2131, requires that the Secretary of Agriculture set standards governing the humane handling, care, treatment, and transportation of animals by research facilities. The standards should ensure that experimental *procedures "ensure* that *animal* pain *and distress* are *minimized,"* and that the investigator considers *"alternatives to any procedure likely to produce pain to or distress in an experimental animal."*
The regulations are found at 9 C.F.R. Ch. 1. Generally, they require each research facility to ensure that its activities *"avoid or minimize discomfort, distress, and pain to the animals."* 9 C.F.R. § 2.31(d)(i). In its annual report, the research facility must certify that each principal investigator has considered alternatives to *"painful procedures,"* (9 C.F.R. § 2.36), defined as *"any procedure that would reasonably be expected to cause more than slight or momentary pain or distress in a human being to which that procedure* was *applied, that is, pain in excess of that caused by injections or other minor procedures."* 9 C.F.R. § 1.1
The Animal Welfare Act thus requires that testing that inflicts pain on the animals is to be carefully scrutinized.

4. Evaluation of Test Feasibility

Applying OECD guidelines and observing relevant provisions for animal welfare, it does not appear that animal testing of spent pulping liquor can reasonably be conducted.

At the pH of this material, primary toxicity is related to the inherent corrosivity of the material. Some of the pertinent OECD test guidelines allow for dilution of the test material used for animal testing. However, in the case of spent pulping liquor, dilution would alter the composition of the material. Thus, the tests of a dilute substance would be addressing a different material - both in composition and because the fundamental corrosive property of the material has been changed. Relevance of testing with such a fundamentally altered substance is highly questionable (and even less potentially applicable than aquatic testing with dilute material). Given the strictures applicable to testing warm-blooded animals, mammalian testing with spent pulping liquor should not be performed.

5. EPA Guidance

The latest guidance from EPA (2000) states: *"In analyzing the adequacy of existing* data, *participants* shall *conduct a thoughtful, qualitative analysis rather than use a rote checklist approach. Participants may conclude that there is sufficient data, given the totality of what is known about a chemical, including human experience, that* certain *endpoints need not be tested."*

Given the high pH and corrosivity of this complex mixture, a thoughtful analysis leads to the conclusion that mammalian testing of spent pulping liquor cannot be justified.

6. In Vitro Genotoxicity Testing

The potential for in vitro genotoxicity will be tested in *Salmonella and a mamallian cell* culture, recognizing that the pH will have to be adjusted in order to ensure survival of the test organisms.

Summary: Given the high pH of spent pulping liquor and the certainty of causing animal suffering should this material be administered in order to conduct the required tests, none of the SIDS human health endpoints involving the use of animals will be undertaken. However, AF&PA will subject spent pulping liquor to in vitro genotoxicity testing.

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Zenz, C, Dickerson, O., and Horvath, E., Eds. Occupational Medicine, 3d edition (1994).

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*  Canadian Centre for Occupational Health and Safety           *
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*** IDENTIFICATION ***

MSDS RECORD NUMBER : 637197
PRODUCT NAME(S) : BLACK LIQUOR
DATE OF MSDS : 1993-08-15

*** MANUFACTURER INFORMATION ***

MANUFACTURER : Canadian Pacific Forest Products Limited
ADDRESS : 2001 Neebing Avenue
Thunder Bay Ontario
Canada P7C 4W3
EMERGENCY TELEPHONE NO. : 807-475-2400 613-996-6666 (CANUTEC)

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DISCLAIMER :
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*** SUPPLIER/DISTRIBUTOR INFORMATION ***

SUPPLIER/DISTRIBUTOR : CANADIAN PACIFIC FOREST PRODUCTS LIMITED
ADDRESS : 1250 Rene-Levesque Boulevard West
Montreal Quebec
Canada H3B 4Y3
Telephone: 514-846-5095

*** MATERIAL SAFETY DATA ***

MATERIAL SAFETY DATA SHEET

BLACK LIQUOR

SECTION 1: PRODUCT INFORMATION

PRODUCT IDENTIFIER: Black liquor
SYNONYMS: Spent kraft pulping liquor, strong black liquor
CHEMICAL NAME: Not applicable
CHEMICAL FORMULA: Mixture, CAS NUMBER 66071-92-9
PRODUCT USE: Ingredient in production of waferboard, adhesives

SECTION 2: HAZARDOUS INGREDIENTS

HAZARDOUS INGREDIENTS*	PERCENT	CAS NUMBER
Sodium carbonate	30-35	487-19-8
LD50:	4,000 mg/kg oral, rat	
LC50:	Not available	
Sodium hydroxide	2-4	1310-73-2
LD50:	40 mg/kg i.p., mouse	
LC50:	Not available	
Sodium sulfide	0,3	16721-80-5
LD50:	30 mg/kg i.p., rat 18 mg/kg subcutaneous, mouse	
LC50:		
Sodium sulfate	< or = 1	7757-82-6
LD50:	Not available	
LC50:	Not available	
Sodium thiosulfate	< or = 1	7772-98-7
LD50:	5200 mg/kg i.p. mouse	
LC50:	Not available	
Silica (quartz)	< or = 1	14808-60-7
LD50:	Not available	
LC50:	Not available	

* Also contains cellulose, hemicellulose, and lignin breakdown products.

SECTION 3: PHYSICAL DATA

PHYSICAL STATE:	Liquid
ODOUR AND APPEARANCE:	Black liquid with rotten egg odour. (Sulphur compounds).
ODOUR THRESHOLD:	Not available
SPECIFIC GRAVITY (water=1):	1,3
VAPOUR PRESSURE:	Not available
VAPOUR DENSITY (air=1):	Not available
EVAPORATION RATE:	Not available
BOILING POINT (deg C):	105-115
FREEZING POINT (deg C):	Not available
pH:	11-13
COEFFICIENT OF WATER/OIL DISTRIBUTION:	Not available
WATER SOLUBILITY (20 deg C):	Not available

SECTION 4: FIRE AND EXPLOSION HAZARDS DATA

CONDITIONS OF FLAMMABILITY:	Not flammable. Will burn at very high temperatures.
MEANS OF EXTINCTION:	Use extinguishing media appropriate to material burning.
FLASH POINT (deg C) & METHOD:	Not applicable
UPPER FLAMMABLE LIMIT (% per volume):	Not applicable

LOWER FLAMMABLE LIMIT (% per volume): Not applicable
AUTO-IGNITION TEMPERATURE (deg C): Not applicable
HAZARDOUS COMBUSTION PRODUCTS: Oxides of carbon and oxides of sulfur
EXPLOSION DATA-SENSITIVITY TO MECHANICAL IMPACT: Not applicable
EXPLOSION DATA-SENSITIVITY TO STATIC DISCHARGE: Not applicable

SECTION 5: REACTIVITY DATA

CHEMICAL INSTABILITY: Not applicable
INCOMPATIBILITY: Aluminum and acids. Contact with acids and oxidizing agents can result in release of potentially lethal concentrations of hydrogen sulfide (H_2S) gas.
HAZARDOUS POLYMERIZATION: Does not occur.
CONDITIONS OF REACTIVITY: Not applicable
HAZARDOUS DECOMPOSITION PRODUCTS: Not applicable

SECTION 6: TOXICOLOGICAL PROPERTIES

ROUTES OF ENTRY: SKIN [ ] INHALATION [X] INGESTION [X]
SKIN CONTACT [X] EYE CONTACT [X]

ACUTE EXPOSURE:
Causes eye and skin irritation and corrosion; respiratory airways irritation; if ingested in large amounts : digestive tract irritation and corrosion, vomiting, diarrhea, circulatory collapsus and death (possible).
Note: Toxicity evaluation of this product was based on sodium carbonate and sodium hydroxide toxicity.
CHRONIC EXPOSURE:
Possibility of dermatitis.
Note: Toxicity evaluation of this product was based on sodium carbonate and sodium hydroxide toxicity.
EXPOSURE LIMITS: Not available
LD50 OF PRODUCT: Not available
LC50 OF PRODUCT: Not available
SENSITISATION TO PRODUCT: Possibility of dermatitis.
CARCINOGENICITY: Not available
TERATOGENICITY: Not available
MUTAGENICITY: Not available
REPRODUCTIVE TOXICITY: Not available
SYNERGISTIC PRODUCTS: Not available

SECTION 7: PREVENTIVE MEASURES

PERSONAL PROTECTIVE EQUIPMENT

GLOVES: Rubber
RESPIRATOR: Use NIOSH/MSHA approved respiratory protection.
EYE PROTECTION: Chemical goggles.
FOOTWEAR: Rubber boots when handling large quantities.
CLOTHING: Rubber suit when handling large quantities.
OTHERS: Remove contaminated clothing immediately and launder before reuse.

ENGINEERING CONTROLS:
Eyewash stations and deluge safety showers nearby Vessels should be washed and ventilated and checked for toxic gas before entry.

LEAK OR SPILL PROCEDURE:
Dike spill with sand or other material. Keep out of sewers or waterways. Pump to approved containers and dispose of in accordance with environmental authorities. Small spills can be washed down with water. Avoid acids as H_2S may be generated.
Consult environmental authorities.
WASTE DISPOSAL:
Consult environmental authorities for proper disposal.
HANDLING PROCEDURE:
Avoid contact with skin. Wear safety glasses. Handle with care.
STORAGE REQUIREMENTS:
Keep in a cool, dry and well-ventilated area. Keep in a tightly closed container and away from acid.
SPECIAL SHIPPING INFORMATION:
TDG identification : PIN-UN/NA number 1760
TDG classification : 8 - corrosive liquids (N.O.S.) - Black Liquor

SECTION 8: FIRST AID MEASURES

EYE CONTACT: Immediate and continuous irrigation with running lukewarm water for at least 30 minutes is imperative. Take care not to rinse contaminated water into the unaffected eye. Call a doctor.
SKIN: Wash off in running water or shower for at least 20 minutes. Remove contaminated clothing immediately and launder before reuse. Call a doctor.
INHALATION: Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a doctor.
INGESTION: Have victim rinse mouth thoroughly with water. DO NOT INDUCE VOMITING. Have victim drink 240 to 300 ml (8 to 10 oz) of water to dilute material in stomach. If vomiting occurs naturally, rinse mouth and repeat administration of water. Never give anything by mouth to an unconscious person. Call a doctor.

SECTION 9: PREPARATION INFORMATION

PREPARED BY: OCCUPATIONAL ENVIRONMENT SERVICES
TELEPHONE NUMBER: (514) 846-5095
DATE OF PREPARATION: August 15, 1993

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* Canadian Centre for Occupational Health and Safety *
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*** IDENTIFICATION ***

MSDS RECORD NUMBER : 168580

PRODUCT NAME(S) : BLACK LIQUOR
DATE OF MSDS : 1989-01-06
CURRENCY NOTE : MSDS Confirmed Current: 1994-06-01

*** MANUFACTURER INFORMATION ***

MANUFACTURER : BUCKEYE CELLULOSE CORPORATION
ADDRESS : POST OFFICE BOX 8407
MEMPHIS TENNESSEE
U.S.A. 38108
EMERGENCY TELEPHONE NO. : 901-320-8100

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*** MATERIAL SAFETY DATA ***

DATE 1/6/89
MATERIAL SAFETY DATA SHEET

SECTION I PRODUCT IDENTIFICATION

TRADE NAME — CAS NO.
BLACK LIQUOR — 66071-92-9

SYNONYMS AND DESCRIPTION
Black liquor is a substance of highly variable alkaline composition produced when wood chips are cooked in the Kraft pulping process. It contains excess pulping chemicals (sodium hydroxide, sodium sulfide), carbonates, sulfates, along with dissolved and degraded cellulose, hemicellulose, and lignins.

SECTION II HAZARDOUS INGREDIENTS

MATERIAL OR COMPONENT		CAS NO.	%
Sodium Hydroxide		1310-73-2	Variable 0.4-4.5
	HAZARD DATA: Irritant, Corrosive		
Sodium Sulfite		7757-82-6	Variable
	HAZARD DATA: Thermal decomposition releases toxic sulfur oxides		
Sodium Sulfide		1313-82-2	Variable
	HAZARD DATA: Reacts with strong acids releasing poisonous hydrogen sulfide.		
Sodium Hydrosulfide		16721-80-5	Variable
	HAZARD DATA: Reacts with strong acids releasing poisonous hydrogen sulfide.		

SECTION III PHYSICAL DATA

BOILING POINT, 760 MM HG	Variable
SPECIFIC GRAVITY (H2O=1)	Variable
VAPOR DENSITY (AIR=1)	Not available
% VOLATILES BY VOL.	Variable
VAPOR PRESSURE (MM HG)	Not available
SOLUBILITY IN H2O% BY WT	Infinite
EVAPORATION RATE (BUTYL ACETATE = 1)	Not available
pH	Typical Range 10-12
APPEARANCE AND ODOR	Black liquid with rotten egg odor.

SECTION IV FIRE AND EXPLOSION HAZARD DATA

FLASH POINT (TEST METHOD) Not available

FLAMMABLE LIMITS IN AIR, % BY VOL. Not available

EXTINGUISHING MEDIA	Not applicable
SPECIAL FIRE FIGHTING PROCEDURES	Not applicable
UNUSUAL FIRE AND EXPLOSION HAZARDS	Not applicable

SECTION V HEALTH AND SAFETY DATA

THRESHOLD LIMIT VALUE None established

EFFECTS OF OVEREXPOSURE

Eye and skin contact can cause serious burns. Possible blindness from eye contact. Will cause serious damage to mouth, throat and stomach if ingested. Inhalation of solution mist may cause upper respiratory tract irritation.

EMERGENCY AND FIRST AID PROCEDURES

EYES:	Flush with water for 15 minutes, get prompt medical attention.
SKIN:	Wash thoroughly with water.
INHALATION:	Remove to fresh air and get medical attention if irritation persists or is severe.
INGESTION:	Do not induce vomiting. Rinse mouth with water. Drink large amounts of water. Get prompt medical attention.

SECTION VI REACTIVITY DATA

STABILITY	UNSTABLE [ ]	CONDITIONS TO AVOID
	STABLE [X]	Not applicable

INCOMPATABILITY (MATERIALS TO AVOID)
Contact with acids can result in release of potentially lethal concentrations of hydrogen sulfide gas.
HAZARDOUS DECOMPOSITION PRODUCTS
Thermal decomposition may produce toxic sulfur oxides.

HAZARDOUS POLYMERIZATION	MAY OCCUR [ ]	CONDITIONS TO AVOID
	WILL NOT OCCUR [X]	Not applicable

SECTION VII DISPOSAL, SPILL OR LEAK PROCEDURES

WASTE DISPOSAL METHOD
Incinerate or dispose of according to local, state and federal laws and regulations for hazardous substances.
STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED
Contains spills. Pump large spills into appropriate container for future use. Absorb small spills in an absorbent material and dispose of as waste. Small spills may be diluted and flushed to an approved treatment system consistent with laws and regulations. Never neutralize spills with acid or divert to acid-containing sewer. Ventilate area. Report as required to the National Emergency Response Center.

SECTION VIII SPECIAL PROTECTION INFORMATION

VENTILATION REQUIREMENTS
General mechanical ventilation is recommended to control odor if stored inside.
SPECIFIC PERSONAL PROTECTIVE EQUIPMENT None required under normal conditions
RESPIRATORY (SPECIFY IN DETAIL) of handling.

EYE	GLOVES
Chemical goggles	Impervious gloves.

OTHER CLOTHING AND EQUIPMENT
Aprons, boots, a rubber suit and face shield may be needed when handling large amounts of this material where splash potential exists or the material is at a high temperature. Eyewash fountain and safety shower.

SECTION IX SPECIAL PRECAUTIONS

PRECAUTIONS TO BE TAKEN IN HANDLING AND STORING
Avoid eye and skin contact. Store away from acids. Avoid discharge into an acid sewer. Spill protect. Check vessels that have contained Black Liquor for hydrogen sulfide before entering. Consult Plant Environmental Control Department regarding waste disposal and spills.
OTHER PRECAUTIONS

SECTION 313 SUPPLIER NOTIFICATION: This product contains 0.4-4.5% sodium hydroxide, CAS No. 1310-73-2 and 0.6-4.5% sodium sulfate, CAS No. 7757-82-6 which are subject to the reporting requirements under SARA TITLE III, Section 313.

ISN: 168580

Attachment E

PSI Incident Report

Blake, Ann

From: Rod Eddy <REddy@pulpmillservices.com>
Sent: Monday, January 27, 2014 12:55 PM
To: Blake, Ann
Cc: mwebb@idealconst.com; Ronnie Marsh
Subject: Crossett Incident report
Attachments: Ideal_Laydown_PSI_Incident-report.pdf

Ms. Blake, Mr. Webb asked that I send you my field report which is attached to this email. The report remains open because I am awaiting the report from the analysis lab.
Please let me know if there is anything else that you need.

Rod Eddy, Director of Safety
Pulpmill Services, Inc.
Reddy@pulpmillservices.com
318-325-4351 - Office
318-535-5024 – Cell
318-387-7143 - Fax

PULPMILL SERVICES, INC

JOB # N/A

INCIDENT REPORT

THIS FORM IS TO BE COMPLETED BY THE SUPERVISOR OR SAFETY COORDINATOR FOR ALL SIGNIFICANT NEAR MISSES AND/OR INJURIES REQUIRING PROFESSIONAL MEDICAL TREATMENT. THIS FORM TO BE RETURNED TO PULPMILL SERVICES SAFETY DIRECTOR WITHIN 8 HRS FOR FATALITIES AND 24 HRS FOR ALL OTHERS.

INJURED INDIVIDUAL:		MILL/SHOP:	
ADDRESS:		AREA WORKING:	
		OCCUPATION:	
PHONE:	CELL:	PPE IN USE: YES	NO
AGE:		LIST ALL PPE:	

INCIDENT INFORMATION:

Date of Incident: 1-9-2014 Time of Incident: Approx, 10:30 AM

Date of Investigation: 1-10-14 through 1-11-14

Name(s) of Investigator(s):

Description of Incident: (Please be brief and factual. Describe what happened. Do not speculate as to causes or corrective actions.)

A Washer Drum being stored at the Ideal Construction Lay-down yard in Crossett, AR was being prepared for transport to the PSI home office in Monroe, LA – Tracy Crocker and Gary Searcy (Slick) were present to perform work. The drum had what they believed to be water in it and they inserted a trash-pump to dispel the water. Shortly after beginning they noticed that diluted black liquor was being pumped out. According to Tracy Crocker's statement, they immediately shut down the pump.

It was raining at the time and Crocker and Slick left the job site not believing there was any issues with the dispensed water/Black liquor.

At approximately 6:30 PM 1-9-14 Ronnie Marsh received a call from Mike Webb who had received a call from the Ashley County Sherriff around 6:00 PM saying that we had something (oil like) running into the yard next to the equipment yard. Containment and clean up (vacuum truck) started around 7:30 PM with sand bags and 3" containment boom. The initial crew left the site at around midnight.

I received a call at 6:50 AM from Ronnie Marsh advising me of the incident. I left the office at around 7:30 and was on site by 8:30. We were back on site at 7:00 Am on 1-10-14 with a crew of 7 to begin the assessment and clean up. I met with Tawana Miller of the Arkansas Department of Emergency Management when I arrived and we discussed the procedures that were being done to abate the incident. I collected water samples from several locations in both yards to have for analysis. There were no signs of residue around the goat pen, the storage shed or the house except at the side of the car port. Sandbagging and initial clean up began and then we were contacted around 1:25 PM I was advised that the next house down also had some residue. We immediately took the vacuum trailer to that yard and vacuumed up all surface residue that we could see. Several 12 – 14 inch deep holes were dug to allow surface drainage and collection. I walked all the property to assess any further migration of the residue and no more was found. Later that day, Donnie Plunks towing and environmental was called to the site to have a certified HAZMAT abatement team on site to direct and assist in clean up. It started raining again at around 3:00 PM Jarrod Gates with DP Towing and Environmental arrived on site at 6:45. He and I walked the affected areas and evaluated the necessary steps to further contain the residue and then to address clean up. The crew left at around midnight. We were back on site at 7:00 AM 1-11-14.

Ms. Miller from OEM arrived at around 7:10 and her and I walked the area to evaluate the containment. The environmental boom materials that had been placed showed VERY LITTLE residue in them. Meaning that most of what was there was discolored water. When Jarrod Gates arrived we collected water samples and tested for Ph levels.

1. City Tap Water as a base line reading was; 7.2 (neutral)
2. Tate Watt driveway: 7.2 (neutral)
3. Drum – too dark to see any color change, Mr. Gates estimated it to be less than 6.8
4. Near Meeks dog Pen – Again too dark to see Mr. Gates again estimated at less than 6.8

All containment materials were left in place since rain was in the forecast.

1-12,13,14 I had Pam Cheek go to site to make sure no further issues had arose. She left cards for the property owners to let them know we were on site these days.

1-15-14 Pam told me she thought it was still to soggy to get the clean-up crew on site so we didn't rut up the yard.

1-15-14 – I arrived on site to evaluate at around 10:30 to make an evaluation of the ground stability for clean-up. I determined it was in good enough condition to begin clean up and contacted the property owners via text Mr. Watt at 11:05 AM and Ms. Meeks at 11:08 AM. Each indicated that was good news. At approximately 3:10 PM this same day, Ms. Meeks contacted me on the office phone to say "her attorney advised her to not let us clean up anything and she should get her own samples for testing. I advised Ronnie Marsh of this development and he contacted me later that evening around 7:10 PM to say that the Arkansas Department of Environmental Quality would be on site Friday morning at 10:00

1-16-2014 Met with Mr. Lamb (ADEQ) and Ms. Miller (AOEM) on site due to a complaint about the spill by Ms. Meeks one of the land owners. MSDS sheets were provided to all parties. While there Mr. Lamb provided a list of laboratories to have samples analyzed and a total of 9 ground samples were collected and marked and one sample from the contents in the drum. The samples we collected later that same day.

1-17-2014 – The collected samples were delivered to American Interplex Corporation Laboratory in Little Rock, AR.

Investigation remains ongoing awaiting the analysis of the samples provided to American Interplex Corporation Lab.

PULPMILL SERVICES, INC

JOB # N/A

FIRST AID PROVIDED: Yes		If Yes, by whom: N/A
POLICE/FIRE/AMBULANCE: Yes		If yes what dept. Ashley County Sherriff
Were photos taken: Yes X	No	If Yes, by whom: Rod Eddy

Attachment F

ADEQ Analytical Report

5301 Northshore Drive
North Little Rock, AR 72118
Telephone: 501-682-0744

Client Report For: Pulp Mill Services Complaint 2014 0263-0268
Attention:
Client Address:

Report Date: February 06, 2014
LAB ID: AR14JAN29-08
Comment:

Approved By:_____ Date:February 06, 2014

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Client: Special Samples
Lab ID: 2014-0263

Client Sample ID: PMS - Drumwasher #1
Collection Date: 1/27/2014 12:37:00 PM
Matrix: Organic

Analyses

Anions by Ion Chromatography — *EPA 9056* — *Batch: 14020608* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
Fluoride	2980	49.8	0.009		mg/Kg
Bromide	<99.6	99.6	.01		mg/Kg
Chloride	4760	199	0.07		mg/Kg
Sulfate	6530	199	0.04		mg/Kg
Weight	0.352				grams
Volume	35				mL
Dilution Factor	10				
Analyzed By	Chad Carrington				
Analysis Date/Time	02/06/14 09:46				

pH — *EPA 150.1* — *Batch: 14013006* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
pH	9.81				SU
Analyzed By	Chad Carrington				
Analysis Date/Time	1/30/2014 15:00				

Percent Solids — *EPA 160.3* — *Batch: 14020601* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
Percent Solids	100	0.2	0.2		%
Analyzed By	Robert Graddy				
Analysis Date/Time	2/05/2014 16:00				

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Client: Special Samples
Lab ID: 2014-0264

Client Sample ID: PMS - Soil #1
Collection Date: 1/27/2014 1:20:00 PM
Matrix: Soil

Analyses

Anions by Ion Chromatography — *EPA 9056* — *Batch: 14020608* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
Fluoride	15.5	5.00	0.009		mg/Kg
Bromide	<9.99	9.99	.01		mg/Kg
Chloride	76.3	20.0	0.07		mg/Kg
Sulfate	352	20.0	0.04		mg/Kg
Weight	0.350				grams
Volume	35				mL
Dilution Factor	1				
Analyzed By	Chad Carrington				
Analysis Date/Time	02/05/14 15:15				

pH-Soil — *EPA 9045D* — *Batch: 14013007* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
pH	8.3				SU
Analyzed By	Chad Carrington				
Analysis Date/Time	1/30/2014 15:00				

Percent Solids — *EPA 160.3* — *Batch: 14020601* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
Percent Solids	64.6	0.2	0.2		%
Analyzed By	Robert Graddy				
Analysis Date/Time	02/05/2014 1600				

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Client: Special Samples
Lab ID: 2014-0265

Client Sample ID: PMS - Soil #2
Collection Date: 1/27/2014 1:36:00 PM
Matrix: Soil

Analyses

Anions by Ion Chromatography — *EPA 9056* — *Batch: 14020608* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
Fluoride	864	25.0	0.009		mg/Kg
Bromide	<49.9	49.9	.01		mg/Kg
Chloride	983	99.8	0.07		mg/Kg
Sulfate	1900	99.8	0.04		mg/Kg
Weight	0.351				grams
Volume	35				mL
Dilution Factor	5				
Analyzed By	Chad Carrington				
Analysis Date/Time	02/05/14 15:30				

pH-Soil — *EPA 9045D* — *Batch: 14013007* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
pH	8.98				SU
Analyzed By	Chad Carrington				
Analysis Date/Time	1/30/2014 15:00				

Percent Solids — *EPA 160.3* — *Batch: 14020601* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
Percent Solids	85.5	0.2	0.2		%
Analyzed By	Robert Graddy				
Analysis Date/Time	02/05/2014 1600				

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Client: Special Samples
Lab ID: 2014-0266

Client Sample ID: PMS - Soil #3 Watt
Collection Date: 1/27/2014 1:45:00 PM
Matrix: Soil

Analyses

Anions by Ion Chromatography *EPA 9056* ***Batch: 14020608 Run: 1***

	Result	Reporting Limit	MDL	Qual	Unit
Fluoride	12.8	4.99	0.009		mg/Kg
Bromide	<9.98	9.98	.01		mg/Kg
Chloride	52.9	20.0	0.07		mg/Kg
Sulfate	157	20.0	0.04		mg/Kg
Weight	0.351				grams
Volume	35				mL
Dilution Factor	1				
Analyzed By	Chad Carrington				
Analysis Date/Time	02/05/14 15:45				

pH-Soil *EPA 9045D* ***Batch: 14013007 Run: 1***

	Result	Reporting Limit	MDL	Qual	Unit
pH	7.06				SU
Analyzed By	Chad Carrington				
Analysis Date/Time	1/30/2014 15:00				

Percent Solids *EPA 160.3* ***Batch: 14020601 Run: 1***

	Result	Reporting Limit	MDL	Qual	Unit
Percent Solids	77	0.2	0.2		%
Analyzed By	Robert Graddy				
Analysis Date/Time	02/05/2014 1600				

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Client: Special Samples
Lab ID: 2014-0267

Client Sample ID: PMS - Soil #4 Meeks
Collection Date: 1/27/2014 2:00:00 PM
Matrix: Soil

Analyses

Anions by Ion Chromatography — *EPA 9056* — *Batch: 14020608* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
Fluoride	6.61	5.00	0.009		mg/Kg
Bromide	<9.99	9.99	.01		mg/Kg
Chloride	40.2	20.0	0.07		mg/Kg
Sulfate	88.4	20.0	0.04		mg/Kg
Weight	0.350				grams
Volume	35				mL
Dilution Factor	1				
Analyzed By	Chad Carrington				
Analysis Date/Time	02/05/14 16:15				

pH-Soil — *EPA 9045D* — *Batch: 14013007* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
pH	5.39				SU
Analyzed By	Chad Carrington				
Analysis Date/Time	1/30/2014 15:00				

Percent Solids — *EPA 160.3* — *Batch: 14020601* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
Percent Solids	68.7	0.2	0.2		%
Analyzed By	Robert Graddy				
Analysis Date/Time	02/05/2014 1600				

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Client: Special Samples
Lab ID: 2014-0268

Client Sample ID: PMS - background
Collection Date: 1/27/2014 3:30:00 PM
Matrix: Soil

Analyses

Anions by Ion Chromatography — *EPA 9056* — *Batch: 14020608* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
Fluoride	<4.98	4.98	0.009		mg/Kg
Bromide	<9.97	9.97	.01		mg/Kg
Chloride	<19.9	19.9	0.07		mg/Kg
Sulfate	24.6	19.9	0.04		mg/Kg
Weight	0.351				grams
Volume	35				mL
Dilution Factor	1				
Analyzed By	Chad Carrington				
Analysis Date/Time	02/05/14 13:31				

pH-Soil — *EPA 9045D* — *Batch: 14013007* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
pH	4.70				SU
Analyzed By	Chad Carrington				
Analysis Date/Time	1/30/2014 15:00				

Percent Solids — *EPA 160.3* — *Batch: 14020601* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
Percent Solids	77.1	0.2	0.2		%
Analyzed By	Robert Graddy				
Analysis Date/Time	02/05/2014 1600				

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Client: Special Samples
Lab ID: 2014-0263

Client Sample ID: PMS - Drumwasher #1
Collection Date: 1/27/2014 12:37:00 PM
Matrix: Organic

Analyses

Metals by ICP *EPA 3051A/EPA 6020A* *Batch: 14020502* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
Aluminum	<200	200	.02		mg/Kg
Antimony	<100	100	0.005		mg/Kg
Arsenic	<10	10	0.0005		mg/Kg
Barium	<100	100	0.002		mg/Kg
Beryllium	<5	5	0.0001		mg/Kg
Cadmium	<10	10	0.0003		mg/Kg
Calcium	<400	400	.04		mg/Kg
Chromium	<10	10	0.0003		mg/Kg
Cobalt	<10	10	0.0005		mg/Kg
Copper	<10	10	0.0005		mg/Kg
Iron	4250	200	.01		mg/Kg
Lead	<10	10	0.0001		mg/Kg
Magnesium	<1000	1000	0.1		mg/Kg
Manganese	16	10	0.0002		mg/Kg
Nickel	<25	25	0.0005		mg/Kg
Potassium	5960	1000	.05		mg/Kg
Selenium	<20	20	0.0005		mg/Kg
Silver	<50	50	0.001		mg/Kg
Sodium	70300	400	.02		mg/Kg
Thallium	<25	25	0.00005		mg/Kg
Vanadium	31.4	25	0.001		mg/Kg
Zinc	<30	30	0.002		mg/Kg
Weight	0.5				grams
Volume	50				mL
Dilution Factor	100				
Analyzed By	Robert Graddy				
Analysis Date/Time	Feb 6 2014 11:24AM				
Prep By					
Prep Date/Time					

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Liitle Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0966

Client: Special Samples
Lab ID: 2014-0264

Client Sample ID: PMS - Soil #1
Collection Date: 1/27/2014 1:20:00 PM
Matrix: Soil

Analyses

Metals by ICP — *EPA 3051A/EPA 6020A* — *Batch: 14020502* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
Antimony	<10	10	0.005		mg/Kg
Arsenic	1.23	1	0.0005		mg/Kg
Barium	51.0	10	0.002		mg/Kg
Beryllium	1.26	0.5	0.0001		mg/Kg
Cadmium	<1	1	0.0003		mg/Kg
Calcium	3460	40	.04		mg/Kg
Chromium	9.56	1	0.0003		mg/Kg
Cobalt	1.39	1	0.0005		mg/Kg
Copper	2.38	1	0.0005		mg/Kg
Lead	8.82	1	0.0001		mg/Kg
Magnesium	2050	100	0.1		mg/Kg
Manganese	520	1	0.0002		mg/Kg
Nickel	3.6	2.5	0.0005		mg/Kg
Potassium	2700	100	.05		mg/Kg
Selenium	<2	2	0.0005		mg/Kg
Silver	<5	5	0.001		mg/Kg
Sodium	8330	40	.02		mg/Kg
Thallium	<2.5	2.5	0.00005		mg/Kg
Vanadium	18.8	2.5	0.001		mg/Kg
Zinc	57.5	3	0.002		mg/Kg
Weight	0.5				grams
Volume	50				mL
Dilution Factor	10				
Analyzed By	Robert Graddy				
Analysis Date/Time	Feb 5 2014 11:13AM				
Prep By					
Prep Date/Time					

Metals by ICP — *EPA 3051A/EPA 6020A* — *Batch: 14020502* *Run: 2*

	Result	Reporting Limit	MDL	Qual	Unit

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Aluminum	14600	200	.02	mg/Kg
Iron	13600	200	.01	mg/Kg
Weight	0.5			grams
Volume	50			mL
Dilution Factor	100			
Analyzed By	Robert Graddy			
Analysis Date/Time	Feb 5 2014 10:27AM			
Prep By				
Prep Date/Time				

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Client: Special Samples
Lab ID: 2014-0265

Client Sample ID: PMS - Soil #2
Collection Date: 1/27/2014 1:36:00 PM
Matrix: Soil

Analyses

Metals by ICP — *EPA 3051A/EPA 6020A* — *Batch: 14020502* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
Antimony	<10	10	0.005		mg/Kg
Arsenic	2.61	1	0.0005		mg/Kg
Barium	114	10	0.002		mg/Kg
Beryllium	0.865	0.5	0.0001		mg/Kg
Cadmium	<1	1	0.0003		mg/Kg
Calcium	4080	40	.04		mg/Kg
Chromium	18.6	1	0.0003		mg/Kg
Cobalt	3.66	1	0.0005		mg/Kg
Copper	9.19	1	0.0005		mg/Kg
Lead	21.4	1	0.0001		mg/Kg
Magnesium	1840	100	0.1		mg/Kg
Manganese	480	1	0.0002		mg/Kg
Nickel	7.8	2.5	0.0005		mg/Kg
Potassium	2850	100	.05		mg/Kg
Selenium	<2	2	0.0005		mg/Kg
Silver	<5	5	0.001		mg/Kg
Sodium	15700	40	.02		mg/Kg
Thallium	<2.5	2.5	0.00005		mg/Kg
Vanadium	26.4	2.5	0.001		mg/Kg
Zinc	287	3	0.002		mg/Kg
Weight	0.5				grams
Volume	50				mL
Dilution Factor	10				
Analyzed By	Robert Graddy				
Analysis Date/Time	Feb 5 2014 11:20AM				
Prep By					
Prep Date/Time					

Metals by ICP — *EPA 3051A/EPA 6020A* — *Batch: 14020502* *Run: 2*

	Result	Reporting Limit	MDL	Qual	Unit

Arkansas Department of Environmental Quality
6301 Northshore Drive
North Liitle Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Aluminum	18200	200	.02	mg/Kg
Iron	14500	200	.01	mg/Kg
Weight	0.5			grams
Volume	50			mL
Dilution Factor	100			
Analyzed By	Robert Graddy			
Analysis Date/Time	Feb 5 2014 10:34AM			
Prep By				
Prep Date/Time				

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Client: Special Samples
Lab ID: 2014-0266

Client Sample ID: PMS - Soil #3 Watt
Collection Date: 1/27/2014 1:45:00 PM
Matrix: Soil

Analyses

Metals by ICP *EPA 3051A/EPA 6020A* *Batch: 14020502* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
Antimony	<10	10	0.005		mg/Kg
Arsenic	6.81	1	0.0005		mg/Kg
Barium	64.8	10	0.002		mg/Kg
Beryllium	0.769	0.5	0.0001		mg/Kg
Cadmium	<1	1	0.0003		mg/Kg
Calcium	3090	40	.04		mg/Kg
Chromium	41.2	1	0.0003		mg/Kg
Cobalt	4.68	1	0.0005		mg/Kg
Copper	18.0	1	0.0005		mg/Kg
Magnesium	1480	100	0.1		mg/Kg
Manganese	570	1	0.0002		mg/Kg
Nickel	8.0	2.5	0.0005		mg/Kg
Potassium	1030	100	.05		mg/Kg
Selenium	<2	2	0.0005		mg/Kg
Silver	<5	5	0.001		mg/Kg
Sodium	1160	40	.02		mg/Kg
Thallium	<2.5	2.5	0.00005		mg/Kg
Vanadium	32.8	2.5	0.001		mg/Kg
Zinc	82	3	0.002		mg/Kg
Weight	0.5				grams
Volume	50				mL
Dilution Factor	10				
Analyzed By	Robert Graddy				
Analysis Date/Time	Feb 5 2014 11:26AM				
Prep By					
Prep Date/Time					

Metals by ICP *EPA 3051A/EPA 6020A* *Batch: 14020502* *Run: 2*

	Result	Reporting Limit	MDL	Qual	Unit
Aluminum	12900	200	.02		mg/Kg

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Liitle Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Iron	19400	200	.01	mg/Kg
Lead	87.2	10	0.0001	mg/Kg
Weight	0.5			grams
Volume	50			mL
Dilution Factor	100			
Analyzed By	Robert Graddy			
Analysis Date/Time	Feb 6 2014 10:40AM			
Prep By				
Prep Date/Time				

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Client: Special Samples
Lab ID: 2014-0267

Client Sample ID: PMS - Soil #4 Meeks
Collection Date: 1/27/2014 2:00:00 PM
Matrix: Soil

Analyses

Metals by ICP — *EPA 3051A/EPA 6020A* — *Batch: 14020502* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
Antimony	<10	10	0.005		mg/Kg
Arsenic	2.62	1	0.0005		mg/Kg
Barium	85.0	10	0.002		mg/Kg
Beryllium	<0.5	0.5	0.0001		mg/Kg
Cadmium	<1	1	0.0003		mg/Kg
Calcium	981	40	.04		mg/Kg
Chromium	20.4	1	0.0003		mg/Kg
Cobalt	3.00	1	0.0005		mg/Kg
Copper	11.0	1	0.0005		mg/Kg
Lead	19.3	1	0.0001		mg/Kg
Magnesium	910	100	0.1		mg/Kg
Manganese	300	1	0.0002		mg/Kg
Nickel	5.6	2.5	0.0005		mg/Kg
Potassium	1120	100	.05		mg/Kg
Selenium	<2	2	0.0005		mg/Kg
Silver	<5	5	0.001		mg/Kg
Sodium	314	40	.02		mg/Kg
Thallium	<2.5	2.5	0.00005		mg/Kg
Vanadium	23.4	2.5	0.001		mg/Kg
Zinc	41.7	3	0.002		mg/Kg
Weight	0.5				grams
Volume	50				mL
Dilution Factor	10				
Analyzed By	Robert Graddy				
Analysis Date/Time	Feb 5 2014 11:33AM				
Prep By					
Prep Date/Time					

Metals by ICP — *EPA 3051A/EPA 6020A* — *Batch: 14020502* *Run: 2*

	Result	Reporting Limit	MDL	Qual	Unit

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Aluminum	12600	200	.02	mg/Kg
Iron	9800	200	.01	mg/Kg
Weight	0.5			grams
Volume	50			mL
Dilution Factor	100			
Analyzed By	Robert Graddy			
Analysis Date/Time	Feb 5 2014 10:47AM			
Prep By				
Prep Date/Time				

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Client: Special Samples
Lab ID: 2014-0268

Client Sample ID: PMS - background
Collection Date: 1/27/2014 3:30:00 PM
Matrix: Soil

Analyses

Metals by ICP — *EPA 3051A/EPA 6020A* — *Batch: 14020502* *Run: 1*

	Result	Reporting Limit	MDL	Qual	Unit
Aluminum	1930	20	.02		mg/Kg
Antimony	<10	10	0.005		mg/Kg
Arsenic	2.82	1	0.0005		mg/Kg
Barium	29.5	10	0.002		mg/Kg
Beryllium	<0.5	0.5	0.0001		mg/Kg
Cadmium	<1	1	0.0003		mg/Kg
Calcium	259	40	.04		mg/Kg
Chromium	15.7	1	0.0003		mg/Kg
Cobalt	21.0	1	0.0005		mg/Kg
Copper	2.77	1	0.0005		mg/Kg
Lead	20.2	1	0.0001		mg/Kg
Magnesium	<100	100	0.1		mg/Kg
Manganese	290	1	0.0002		mg/Kg
Nickel	9.7	2.5	0.0005		mg/Kg
Potassium	<100	100	.05		mg/Kg
Selenium	<2	2	0.0005		mg/Kg
Silver	<5	5	0.001		mg/Kg
Sodium	<40	40	.02		mg/Kg
Thallium	<2.5	2.5	0.00005		mg/Kg
Vanadium	16.9	2.5	0.001		mg/Kg
Zinc	12.5	3	0.002		mg/Kg
Weight	0.5				grams
Volume	50				mL
Dilution Factor	10				
Analyzed By	Robert Graddy				
Analysis Date/Time	Feb 5 2014 11:39AM				
Prep By					
Prep Date/Time					

Metals by ICP — *EPA 3051A/EPA 6020A* — *Batch: 14020502* *Run: 2*

	Result	Reporting	MDL	Qual	Unit

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Liitle Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

		Limit		
Iron	8740	200	.01	mg/Kg
Weight	0.5			grams
Volume	50			mL
Dilution Factor	100			
Analyzed By	Robert Graddy			
Analysis Date/Time	Feb 5 2014 10:53AM			
Prep By				
Prep Date/Time				

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Analytical Quality Control Results Report

Batch: 14013006	pH - water
LCS	LIMS ID: 14013006-LCS-01

pH - water LCS *Run: 1*

Parameter	Result	DL	RL	Accuracy Control	Precision Control
pH (% Recovery)	99.5 %			95 - 105	
Analyzed By	Chad Carrington				
Analysis Date/Time	1/30/2014 15:00				

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Analytical Quality Control Results Report

Batch: 14013007	pH - soil
LCS	LIMS ID: 14013007-LCS-01

pH - soil LCS — Run: 1

Parameter	Result	DL	RL	Accuracy Control	Precision Control
pH (% Recovery)	99.5 %			95 - 105	
Analyzed By	Chad Carrington				
Analysis Date/Time	1/30/2014 15:00				

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Analytical Quality Control Results Report

Batch: 14020608 — **Anions - soil**

PMS - Drumwasher #1 — LIMS ID: 2014-0263

Anions - soil DUP — Run: 1

Parameter	Result	DL	RL	Accuracy Control	Precision Control
Fluoride	2970 mg/Kg	8.93	49.6		
Fluoride (RPD)	0.3 %				0 - 20
Bromide	<99.2 mg/Kg	9.92	99.2		
Bromide (RPD)	0 %				0 - 20
Chloride (RPD)	0.5 %				0 - 20
Chloride	4730 mg/Kg	69.5	198		
Sulfate (RPD)	1.2 %				0 - 20
Sulfate	6450 mg/Kg	39.7	198		
Weight	0 grams				
Volume	35 mL				
Dilution Factor	10				
Analyzed By	Chad Carrington				
Analysis Date/Time	02/06/14 10:01				

PMS - Soil #3 Watt — LIMS ID: 2014-0266

Anions - soil DUP — Run: 1

Parameter	Result	DL	RL	Accuracy Control	Precision Control
Fluoride	13.5 mg/Kg	0.9	4.98		
Fluoride (RPD)	5.1 %				0 - 20
Bromide (RPD)	0 %				0 - 20
Bromide	<9.97 mg/Kg	1	9.97		
Chloride (RPD)	0.3 %				0 - 20
Chloride	63.1 mg/Kg	6.98	19.9		
Sulfate	154 mg/Kg	3.99	19.9		
Sulfate (RPD)	1.6 %				0 - 20
Weight	0 grams				
Volume	35 mL				
Dilution Factor	1				
Analyzed By	Chad Carrington				
Analysis Date/Time	02/05/14 16:00				

MB — LIMS ID: 14020608-MB-01

Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

Laboratory Contact: Jeff Ruehr
Ruehr@adeq.state.ar.us
501-682-0955

Anions - soil MB ***Run: 1***

Parameter	*Result*	*DL*	*RL*	*Accuracy Control*	*Precision Control*
Fluoride	<0.05 mg/Kg	0	0.05		
Bromide	<0.1 mg/Kg	0.01	0.1		
Chloride	<0.2 mg/Kg	0.07	0.2		
Sulfate	<0.2 mg/Kg	0.04	0.2		
Weight	1 grams				
Volume	1 mL				
Dilution Factor	1				
Analyzed By	Chad Carrington				
Analysis Date/Time	02/05/14 11:49				

LCS ***LIMS ID: 14020608-LCS-01***

Anions - soil LCS ***Run: 1***

Parameter	*Result*	*DL*	*RL*	*Accuracy Control*	*Precision Control*
Fluoride (% Recovery)	103 %			90 - 110	
Bromide (% Recovery)	105 %			90 - 110	
Chloride (% Recovery)	106 %			90 - 110	
Sulfate (% Recovery)	105 %			90 - 110	
Weight	1 grams				
Volume	1 mL				
Dilution Factor	1				
Analyzed By	Chad Carrington				
Analysis Date/Time	02/05/14 12:04				

ADEQ
ARKANSAS
Department of Environmental Quality

Arkansas Department of Environmental Quality
Chain of Custody
for Compliance or Enforcement Samples

Facility, Project, Complaint Name, Property Owner: Pulp Mill Services/ ~~GP Grossett~~ JAP 1/28/14

AFIN #/ County:

Printed Names of Sampler(s): Chris Krok/Ann Blake

ADEQ Division or Other (Describe): HWD

- ☐ CSI
- ☐ Other Compliance
- ☑ Complaint
- ☐ Fish Kill
- ☐ Other (describe)

Media Code: W = water; G = groundwater; L = liquid (not water); S = soil or solid; E = edible tissue; F = whole fish; B = other

Preservation Code: A = Cool to 4°C; B = Sulfuric Acid; C = Nitric Acid; D = NaOH; E = Sodium Thiosulfate; F = Other (specify)

Sample ID	Date Collected mm/dd/yy	Time Collected hh:mm	Sample Characteristics: Grab	Composite	No. of Containers	Preservation Code	Media Code	Parameters Requested: semivolatile	anions	Total Metals	PH	Sample Remarks	Latitude (dd.ddddd)	Longitude (dd.ddddd)	Lab #
Drumwasher #1	1/27/14	12:37	X		7	A	L	X	X	X	X	if metals high then TCLP			
Soil #1	1/27/14	13:20	X		2	A	S	X	X	X	X				
Soil #2	1/27/14	13:30	X		1	A	S	X	X	X	X				
Soil #3 - Watt	1/27/14	13:45	X		1	A	S	X	X	X	X				
Soil #4 - Meeks	1/27/14	14:00	X		1	A	S	X	X	X	X				
Soil - background	1/27/14	15:30	X		1	A	S	X	X	X	X				

Relinquished by	Date	Time	Received by	Date	Time
Relinquished to Laboratory by	**Date**	**Time**	**Received for Laboratory by**	**Date**	**Time**
Ann Blake	1/28/14	9:04 AM	Jeff Rush	1/28/14	9:04

Notes: Liquids are syrupy & heavy sludge

Use back for additional notes and remarks

Attachment G

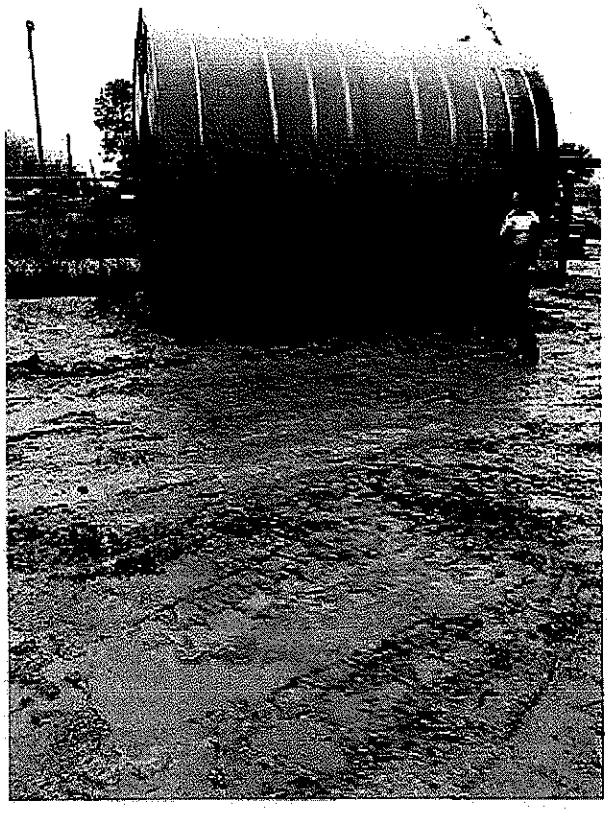
Photographic Log

Photographs
Taken by
Rod Eddy of PSI

Attachment G

Photographic Log

Photographs
taken by
Rod Eddy of PSI
2/4/14 @ 2:37 p.m.



Attachment H

PSI E-mails

Blake, Ann

From: Rod Eddy <REddy@pulpmillservices.com>
Sent: Wednesday, January 29, 2014 3:58 PM
To: Blake, Ann
Cc: Ronnie Marsh
Subject: RE: American Interplex Report

Ms. Blake,

I will get the pictures put on a flash drive and try to get out in tomorrow's mail. I will be out delivering the lab reports to the property owners but will try to have it done before the end of the day.
As for the name of the vacuum truck company, I am getting that information from the office and will forward as soon as I have it. The contents that were collected by the vacuum truck are in a collection tank on site so it has not been disposed of yet.
Not sure where Mr. Lamb came up with the drum coming out of GP. The drum did come from the Gilman Paper Mill which is no longer in business and we do not have any paperwork on it.
The tarped material was taken to the Ashley County Solid Waste facility 205 East Jefferson, Hamburg, AR Ticket number 121649 I will get a copy of that scanned and will send that to you.

Rod Eddy, Director of Safety
Pulpmill Services, Inc.
Reddy@pulpmillservices.com
318-325-4351 - Office
318-535-5024 – Cell
318-387-7143 - Fax

From: Blake, Ann [mailto:BLAKE@adeq.state.ar.us]
Sent: Wednesday, January 29, 2014 3:45 PM
To: Rod Eddy
Subject: RE: American Interplex Report

Ok, that would be fine.

Ann Blake
Arkansas Department of Environmental Quality
5301 Northshore Drive
North Little Rock, AR 72118

As for the other questions, you can e-mail me that information.
Thanks in advance.

From: Rod Eddy [mailto:REddy@pulpmillservices.com]
Sent: Wednesday, January 29, 2014 3:39 PM
To: Blake, Ann
Subject: RE: American Interplex Report

Ms. Blake,

Yes I have several pictures taken at different intervals. The camera was new and the date and time stamp did not record.
The picture file is over 400 MB so I am wondering if it would be better for me to load them to a flash drive and send to you?
Please give me your mailing address.

Rod Eddy, Director of Safety
Pulpmill Services, Inc.
Reddy@pulpmillservices.com
318-325-4351 - Office
318-535-5024 – Cell
318-387-7143 - Fax

From: Blake, Ann [mailto:BLAKE@adeq.state.ar.us]
Sent: Wednesday, January 29, 2014 3:07 PM
To: Rod Eddy; Lamb, John; ashleyoem@sbcglobal.net
Cc: Ronnie Marsh; Krou, Christopher
Subject: RE: American Interplex Report

Mr. Eddy,

On the phone, you mentioned that photographs were taken. Can you please send me the photographs taken and the name of the company used to vacuum up the black liquor. Also I would like to find out where the material in the vacuum truck went for disposal and a copy of the bill of lading. Also, I'd like to find out where the tarped material in the red pull trailer was disposed of as well and a copy of any paperwork to follow. According to John Lamb, he indicated that you had told him the drum washer had been taken out of the Georgia Pacific Crossett Paper Mill some 14 years prior. If this is correct, would you have any paper trail to verify that it came from them? Jim Cutbirth of GP indicated that it came from a Gilman Paper Mill?

From: Rod Eddy [mailto:REddy@pulpmillservices.com]
Sent: Wednesday, January 29, 2014 2:11 PM
To: Blake, Ann; Lamb, John; ashleyoem@sbcglobal.net
Cc: Ronnie Marsh
Subject: American Interplex Report

Ms. Blake, Mr. Lamb, Ms. Miller;
Please find attached the results from American Interplex Corporation Lab regarding the "black liquor" incident on Hancock Rd. Crossett, AR
I added the sample locations on the attached report for reference. If you would like a copy without the reference notes, just let me know and I can forward that to you.
In the report is a copy of all transmittal documents delivered to American Interplex at time of delivery to them for analysis. (MSDS and American Forest & Paper Association report AR 201-12936)
Also incorporated in the attachment is an email from Mr. Bradford with American Interplex today in response to a question I asked him to address.
If I can provide you anything else at this time, please let me know.
I will be hand delivering a copy of this report and correspondence to the home owners tomorrow morning.

Respectfully,

Rod Eddy, Director of Safety
Pulpmill Services, Inc.
Reddy@pulpmillservices.com
318-325-4351 - Office
318-535-5024 – Cell
318-387-7143 - Fax

Blake, Ann

From: Blake, Ann
Sent: Friday, January 31, 2014 10:51 AM
To: 'Rod Eddy'
Cc: Ronnie Marsh; Wilson, Penny
Subject: RE: requested information

Tracking:	**Recipient**	**Delivery**	**Read**
	'Rod Eddy'		
	Ronnie Marsh		
	Wilson, Penny	Delivered: 1/31/2014 10:51 AM	Read: 1/31/2014 10:52 AM

Thank you for the information supplied via email and photo's you have sent out. However, each individual generator of a solid waste is responsible for evaluating their own waste and making a hazardous waste determination from that evaluation. The RCRA regulations place the burden on the generator to determine whether a solid waste is a hazardous waste – ADEQ cannot make the determination for you. As to your question below, our lab has not yet finished analyzing the ADEQ samples taken on Monday 1/27/14.

From: Rod Eddy [mailto:REddy@pulpmillservices.com]
Sent: Thursday, January 30, 2014 1:34 PM
To: Blake, Ann
Cc: Ronnie Marsh
Subject: requested information

Ms. Blake;
The company we used with the vacuum truck was;
A&E Environmental
1675 Marais Saline Rd. Crossett, Ar. 71635
870-304-6586

And as I stated yesterday, everything that was vacuumed is in the tank on site.

For your information, I met with the property owners this morning and hand delivered the lab results to them.
Has your office concluded your sample testing? We will of course rely on your direction once you have concluded that portion of your investigation along with the lab results we provided you a copy of yesterday and then we will look to you and your office for further (if any) direction.
The flash drive with the pictures, went out in todays mail.

Respectfully,

Rod Eddy, Director of Safety
Pulpmill Services, Inc.
Reddy@pulpmillservices.com
318-325-4351 - Office
318-535-5024 – Cell
318-387-7143 - Fax

Blake, Ann

From: Wilson, Penny
Sent: Wednesday, February 05, 2014 8:12 AM
To: Blake, Ann
Subject: FW: Cleanup
Attachments: IMG_20140203_204251.jpg; IMG_20140204_112410.jpg; IMG_20140204_111547.jpg

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From: VanDerhoff, Dean
Sent: Tuesday, February 04, 2014 3:18 PM
To: Hynum, Tammie; Wilson, Penny
Subject: FW: Cleanup

-----Original Message-----
From: Rod Eddy [REddy@pulpmillservices.com]
Sent: Tuesday, February 04, 2014 02:37 PM Central Standard Time
To: VanDerhoff, Dean
Subject: RE: Cleanup

Dean been raining all day but we are working. We have removed all the affected soil in the yard where the drum is. We have moved the drum to remove the soil from underneath it also. Samples have been sent to American Interplex to determine if it can be taken to a class 1 landfill. Our roll off containers have been requested they want the determination from the lab before they bring them out. The soil has also been removed from the yard next door between the fence and driveway.
Respectfully, Rod Eddy

"VanDerhoff, Dean" <VANDERHOFF@adeq.state.ar.us> wrote:

Got them. Thanks.

-----Original Message-----
From: Rod Eddy [REddy@pulpmillservices.com]
Sent: Monday, February 03, 2014 04:58 PM Central Standard Time
To: VanDerhoff, Dean
Subject: Re: Cleanup

I had 3 pictures returned. Trying again.

"VanDerhoff, Dean" <VANDERHOFF@adeq.state.ar.us> wrote:

I need you to call me or my chief at 5016820831 immediately.